Examinations for Association or Origin

1 Introduction

Manufactured goods and the items (e.g., fragments) derived from them bear characteristics indicative of their processing history and subsequent use in service. These characteristics include the specific alloy type, the specific composition of a particular alloy, the physical dimensions, fabrication marks from the forming process, the presence of welds, the microstructure, damage suffered in service such as fracture, wear, thermal damage, and numerous others. These characteristics can be used to determine a potential source of an item or distinguish among items which are nominally of the same class. For example, two pipe sections from different sources can be distinguished based on their diameter, method of fabrication, alloy content, the nature of plating materials on them, the presence or absence of fabrication marks and so on. Similarly, two sections cut from a common length of pipe would be expected to be indistinguishable in all of these characteristics.

2 Scope

This document applies to caseworking personnel who perform metallurgy analyses. In metallurgy, there is an extremely wide variety of components, metals, treatments, fabrication techniques, service abuse, types of failure and damage, applications, and exposure environments that are variables affecting evidentiary materials. The following procedure outlines the basic analyses most commonly performed to examine an item for association or origin.

3 Principle

Items can be examined to potentially identify their source or origin of manufacture. Compositional and physical characteristics can often be used to distinguish between places of manufacture and possibly even between different production lots by the same manufacturer. These characteristics can include chemical composition, physical marks imparted by fabrication tooling, dimensions and other design and fabrication considerations. Often one or more exemplars that share some characteristic(s) with the evidentiary items can provide useful information.

Items suspected of being from common sources are compared against each other in their relevant compositional and physical characteristics using those techniques which are most appropriate. These typically include visual and microscopic examination of the surfaces and other characteristics, dimensional measurements, evaluation of the fabrication characteristics, and compositional analysis of the items. Two items which are not distinguished from each other on these bases are considered to demonstrate an association and possibly a common origin.

4 Specimens

Nearly any metal object and many nonmetallic objects can be examined using the steps outlined in this procedure.

5 Equipment/Materials/Reagents

A list of items commonly used in these examinations follows. Not every item is used for all association and origin investigations. The instrumentation and equipment to be employed will depend on the nature of the item(s) to be examined and compared. When an instrument marked with an asterisk is used, see the appropriate Chemistry Unit (CU) Metallurgy standard operating procedure (SOP) for additional supplies (see section 15 References).

a. Photography equipment for macro- and micro-documentation

b. Observation enhancing tools, such as:
   i. borescope, magnifying glass, jewelers' loupe
   ii. visible light microscopes (stereomicroscope, digital microscope, comparison microscope)
   iii. scanning electron microscope (SEM)

c. Radiography system*

d. Measurement tools, such as:
   i. micrometers, calipers, measuring tape
   ii. optical measuring microscope (e.g., SmartScope FOV*)
   iii. balances
   iv. magnet

e. Miscellaneous hand tools

f. Certified reference materials and calibration standards as needed

g. Digital multimeter

h. Specimen cleaning and protection equipment and materials:
   i. compressed air
   ii. lint free wipes
   iii. cleaning brushes
   iv. cellulose acetate replication tape
   v. EvapoRust™ rust remover
   vi. Solvents: water, alcohol, etc.
   vii. ultrasonic cleaner
   viii. desiccant
   ix. vacuum chamber

i. Compositional analysis instruments:
    i. Energy dispersive x-ray fluorescence spectrometer (EDXRF)*
    ii. Spark discharge-in-argon optical emission spectrometer (SDAR-OES)*
    iii. Scanning electron microscope with energy dispersive x-ray spectrometer (SEM/EDS)

j. Metallographic sample preparation and examination equipment*

k. Non-destructive testing equipment, such as:
    i. magnetic particle inspection equipment
    ii. liquid dye penetrant (LDP) and developer
    iii. ultrasonic inspection equipment

l. Mechanical testing instruments, such as:
    i. Hardness* and microhardness* testers
    ii. Tensile*, torsion, fatigue, impact and wear testers

6 Standards and Controls

The standards and control samples used in this procedure will depend on the specific analytic methods employed and the nature of the item under analysis. Any instrument used in this procedure will employ such standards as required under its specific standard operating procedure. Exemplars for evidentiary items may be obtained and examined to establish the expected variability of manufactured characteristics.

7 Sampling

Visual examinations are performed on every item examined under this protocol. Further testing is based on the suitability of individual items, or portions of items, for relevant examination techniques. Case notes will describe which examinations were performed on which items. If initial examinations reveal that an analyzed characteristic may vary on a single item, the means of selecting a location to test the characteristic will be noted in the case file.

If an item contains a large number of visually indistinguishable objects that are suitable for one analysis technique, a subset may be selected for testing by (1) non-statistical or (2) statistical means. Any sampling plan and corresponding procedure used will be recorded in case notes.

(1) For non-statistical specimen selection, the report will attribute the measured characteristic only to the specimen(s) tested. This can be facilitated by sub-dividing the evidence and reporting the specific analysis results for the sub-divided portion only.

(2) If a sampling plan will be used to make an inference about the entire set of visually similar items, then the plan will be based on a statistically valid approach. A hypergeometric distribution can be used to describe the probability of encountering deviations within a set of items when not

every item is tested. (See Appendix A.) Appendix A assumes that all results are consistent. If inconsistent results are encountered, metallurgy conclusions regarding that characteristic will be limited to the specimens tested.

8 Procedure

The following steps and/or tests are not required in every situation and will vary depending on circumstances and the evidence. Additionally, the sequence below serves only as a general guideline, and the examinations selected should be established by the facts and circumstances of the case. Data gathered during examinations will be included in the case notes. This procedure will not be taken as a substitute for sound engineering judgment.

a. Perform a preliminary visual and low magnification microscopic evaluation of the item(s) to evaluate the fabrication method(s); fracture and/or damage morphology; materials processing characteristics; material transfer; and any other characteristics deemed to be of value.

b. Photograph submitted or in-situ items in the “as-received condition” (ARC). Additional photography should be conducted during the metallurgical examinations to record any features or characteristics upon which a conclusion is likely to be based. Whenever practicable, include a scale in the photograph or apply a verified micron marker to the photograph.

c. Evaluate the physical properties of the items by measuring appropriate features, such as dimensions, mass and magnetic response.

d. Perform a radiographic examination of the specimens looking for internal structure(s), contaminants, defects, and any other appropriate characteristics suitable for evaluation by this technique.

e. Conduct visual and low power magnification examinations for characteristics of shape, size, material(s), fabrication characteristics/marks, anomalies, processing characteristics, modifications for service or other post-purchase use, service abuse, non-service abuse, characteristics of environmental interaction, existence of fractures and/or damage, manner of separation or failure, exogenous residues/deposits (composition and manner of deposition), and any other characteristics of value.

f. Perform higher magnification examinations and/or comparisons of fabrication and materials processing characteristics, morphological features, fracture surface evaluations, exogenous deposit characterization, damage site evaluation, and any other higher magnification examination deemed appropriate.

g. Microscopic inspections may be augmented with scanning electron microscopy (SEM). Typically, this should be done when optical instruments are unable to resolve sample features which the examiner deems are important in reaching a conclusion.

h. Assess the characteristics of environmental interaction(s) as appropriate for the determination(s) requested. Apparent differences in corrosion behavior should be reconciled with the facts or feasible explanations of material behavior and/or environmental parameters.

i. Perform qualitative, semi-quantitative, or quantitative compositional analysis of any materials observed during examinations under this protocol which may assist in associating specimens and characteristics and/or determining possible origin. Samples or sections may be taken from the items for chemical analyses of coating(s), substrate material(s), corrosion product(s), deposits, contaminants, or any other material relevant to the determination(s) requested.

j. The above examinations may be augmented by various inspection and testing techniques, including non-destructive inspection, mechanical property testing (i.e., hardness, tensile, impact testing) and metallography.

k. Any destructive testing should be performed with regard to minimizing material loss and retaining informative features.

l. Report findings after evaluation of all gathered data.

9 Instrumental Conditions

For instruments that require verification, standardization or energy adjustment, a copy of the appropriate record(s) will be included in the case notes.

9.1 Analytical Instruments (for SEM/EDS see 9.3)

For each instrument noted (*) in 5 Equipment/Materials/Reagents, follow the appropriate CU Metallurgy SOP (see section 15 References).

9.2 Supporting Equipment

The following additional instrumental conditions also will be applied:

a. Macro- and micro-photographs will contain a reference scale whenever feasible, however these are included for general reference, and measurements will not be made from the images. Micron markers that are automatically generated by camera or microscope software are to be considered approximate and also will not be used to measure features within the image unless the marker is verified against a calibrated scale.

b. When possible, cutting and grinding operations will be lubricated to prevent overheating that can change the metallurgical characteristics of the specimen. If lubrication is not possible, the metallurgical changes imparted by the process must be considered during analysis.

c. The following instruments will be verified according to the appropriate CU Instrument Operations Systems Support (IOSS) SOP (see 15 References) prior to their first use to acquire case data on any given day:
    i. traceable micrometers/calipers
    ii. traceable balances

9.3 SEM/EDS

Compositional analysis by SEM/EDS will be conducted as follows:

a. Prior to the first use to acquire case data on any given day, run the instrument performance verification routine according to the appropriate IOSS SOP (see 15 References). File one copy with the instrument performance records.

b. Prepare and insert the specimen(s) ensuring electrical continuity with the sample stage.

c. Adjust the instrument conditions to image the region of interest for analysis. Backscattered electron imaging can be helpful to locate features that differ in mean atomic number from their surroundings.

d. Acquisition duration will depend on the conditions chosen and the sample area exposed to the incident beam. The acquisition time can be extended to optimize spectrum clarity or shortened to enhance collection efficiency based on the case requirements.

e. Label the elemental peaks on the acquired spectrum, considering peak shapes and energy positions, the relative heights of adjacent peaks and system-generated peaks. Many SEM/EDS systems have software that can accurately identify the escape and sum peaks in a spectrum. The peak identification system resident in the instrument software can be augmented by analyzing CRMs of similar composition to the specimen of interest.

f. Ensure the instrument identification and the operating parameters are recorded on the printed spectra or elsewhere in the case notes.

10 Decision Criteria

A conclusion that a particular item is from a particular origin, or may be associated with another known item is based on a series of direct comparisons with that known item. Normally, all examinations conducted on known and questioned items must yield comparable results if an association is to be reported. However, observed differences which can reasonably be explained within the established factual framework of a particular case do not preclude an association from being made. Conclusions will be expressed in reports and testimony according to current FBI Laboratory requirements (see section 15 References).

The results of examinations for association can be expressed as ‘fracture fit’, ‘inclusion’, ‘exclusion’, or ‘inconclusive’ conclusions:

'Fracture fit' is an examiner's conclusion that two or more metallurgy items or materials were once part of the same object. This conclusion is an examiner's decision that two or more metallurgy items or materials show sufficient correspondence between their observed characteristics to indicate that they once comprised a single object and insufficient disagreement between their observed characteristics to conclude that they originated from different objects. This conclusion can only be reached when portions of two or more metallurgy items or materials physically fit together.

'Inclusion' is an examiner's conclusion that two or more metallurgy items or materials could have originated from the same source or process. An examiner may conclude that two or more items or materials originated either from the same metallurgy source or process or from another source or process that is substantially similar to the examined items or materials in all observed characteristics. An item or material may be included within a broad general population of items or materials (such as those that are mass-produced), or to a less frequently encountered population of items or materials, based on their physical and chemical characteristics. The basis for an 'inclusion' conclusion is an examiner's decision that two or more items or materials exhibit substantially similar observed characteristics with no unexplainable differences.

'Exclusion' is an examiner's conclusion that the metallurgy items or materials could not have originated from the same source or process. The basis for an 'exclusion' conclusion is an examiner's decision that two or more items or materials exhibit substantially dissimilar observed characteristics that would not be expected from items or materials that originated from the same source or process.

'Inconclusive' is an examiner's conclusion that no determination can be reached as to whether two or more metallurgy items or materials could have originated from (or be excluded as originating from) the same source or process. The basis for an 'inconclusive' conclusion is the examiner's decision that there is insufficient quantity and/or quality of observed characteristics to determine whether two or more items or materials could have originated from the same process (or be excluded as originating from the same process.)

11 Calculations

In most instances, no calculations are required to perform this procedure. Calculations associated with the use of particular instruments will be found in the appropriate SOP.

Where quantitative data from two specimens are being compared, a pooled, two-tailed Student's t-test statistic of the sample means is typically used for the comparison. Two samples are deemed to be "indistinguishable" in the property under consideration if the two samples differ by less than the preselected critical t value ($t_{critical}$). The critical t values are typically chosen so that an overall value of $\alpha = 0.05$ can be achieved for the analysis and are determined by the degrees of freedom associated with the measurement. An $\alpha = 0.05$ means there is a 5.0% chance of incorrectly rejecting a match between two samples when one actually exists.

To perform this test, the means and variances of each sample are determined as follows:

The mean value: $\bar{x}_a = \frac{\sum_{i=1}^{n_a} x_i}{n_a}$ where: $\bar{x}_a$ is the average value of the measurements on sample "a", $\sum x_i$ is the sum of the individual measurements and n_a is the number of measurements made on that sample.

The variance of the individual measurement values from sample "a" is given by:

$$s_a^2 = \frac{\sum_{i=1}^{n_a} (x_i - \bar{x})^2}{n_a - 1}$$

The mean and variance of the data from sample "b" are calculated in the analogous manner.

The pooled sample variance is then calculated as: $s_p^2 = \frac{(n_a - 1)s_a^2 + (n_b - 1)s_b^2}{(n_a + n_b - 2)}$

A standard two-tailed statistical test of the two sample means is performed.

If $\left| \frac{(\bar{x}_a - \bar{x}_b)}{\left(\sqrt{s_p^2 (\frac{1}{n_a} + \frac{1}{n_b})}\right)} \right| > t_{critical}$, the samples are concluded to have a statistically significant difference. If not, the samples are concluded to be indistinguishable.

In general, the number of individual measurements required per sample is determined by the population data distribution. If the sample populations are known to be or can reasonably be assumed to be normally distributed (Gaussian), as few as three measurements per sample can be used to compare the results. In the majority of instances where the measurement populations are not normally distributed, 5-10 measurements per sample will result in sample means which are approximately normal and will be adequate for the comparison outlined above. For heavily skewed population distributions, a minimum of 30 measurements per sample may be required to achieve this. Heavily skewed data distributions will normally be detectable on inspection of the sample data. Statistical tests also exist for determining whether data are Gaussian or non-Gaussian and can be employed as they are needed. Commonly, a normal probability plot is constructed for this purpose using statistical software packages such as MINITAB or equivalent.

12 Measurement Uncertainty

When gathered, quantitative data are generally used for comparative purposes. Expanded uncertainty should not be used for these inter-comparisons because it increases the probability

two samples will appear to be analytically indistinguishable and therefore increases the likelihood of type II errors (false inclusion).

In the event that it is necessary to calculate the expanded uncertainty of a measurement, it will be done in accord with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*. Instrumental measurement uncertainty is addressed in the individual instrument SOPs and will be calculated and reported when appropriate. Each time measurement uncertainty is calculated and reported, the repeatability component(s) will be updated. Often the variation present in a part production run, or allowed in a part specification, is substantially larger than the uncertainty contribution from the measuring instrument. In these cases, instrument measurement uncertainties will not be reported because they are considered negligible.

13 Limitations

The limitations of a particular analysis (if any) are determined by the type of sample(s) being analyzed, the condition of the samples, the specific determinations being made, and the specific examinations required in the situation under consideration and cannot therefore be predicted within this protocol but will be reported when appropriate. See also section 10 Decision Criteria.

14 Safety

a. Wear an x-ray film badge or dosimeter when operating instruments that generate x-rays. The instruments have protective enclosures and internal safety interlocks to prevent inadvertent x-ray radiation exposure. Never bypass or disable safety interlocks on instruments.

b. Wear personal protective gear and use engineering controls that are appropriate for the task being performed (e.g., safety glasses when cutting and chemical fume hood when etching). Electrical or mechanical hazards may require special precautions (e.g., grounding to prevent electric shock or wearing a face guard to prevent impact from flying debris.) Review instrument SOPs and pertinent material Safety Data Sheets (SDS) prior to conducting examinations. If additional guidance is required, contact the Laboratory Health and Safety Group.

15 References

ASM International Handbook Committee, *ASM Handbook, Volume 8 - Mechanical Testing and Evaluation*, ASM International 2000, or latest revision

ASM International Handbook Committee, *ASM Handbook, Volume10-Materials Characterization, ASM International* 1992, or latest revision

Oberg, E., Jones, F. D., Horton, H. L., and Ryffel, H. H., *Machinery's Handbook*, 25th edition, Industrial Press Inc. 1996

Encyclopedia of Forensic Sciences, Editor-in-Chief Siegel, J.A., Academic Press 2013

Forensic Science Handbook, 2nd Edition, Editor Saferstein, R., Prentice-Hall Press 2002

Anderson, R. C., *Inspection of Metals, Volume I: Visual Examination*, American Society for Metals 1983

Milton, J. S. and Arnold, J. C., *Introduction to Probability and Statistics - Principles and Applications for Engineering and Computer Sciences, Fourth Edition*, McGraw-Hill Higher Education, 2003

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

General Approach to Report Writing in Metallurgy, Metallurgy Manual Metal 900, Chemistry Unit, latest revision

Chemistry Unit (CU) FBI-Approved Standards for Scientific Testimony and Report Language for Metallurgy, Metallurgy Manual Metal 901, Chemistry Unit, latest revision

Department of Justice Uniform Language for Testimony and Reports for the Forensic Metallurgy Discipline, latest revision

Digital Radiography, Metallurgy Manual Metal 303, Chemistry Unit, latest revision

Operation of the SmartScope FOV Video Measurement System, Metallurgy Manual Metal 302, Chemistry Unit, latest revision

Compositional Analysis by Energy Dispersive X-Ray Fluorescence Spectrometry (EDXRF), Metallurgy Manual Metal 500, Chemistry Unit, latest revision

Compositional Analysis by Spark Discharge in Argon Optical Emission Spectroscopy (SDAR-OES), Metallurgy Manual Metal 400, Chemistry Unit, latest revision

Metallographic Examinations, Metallurgy Manual Metal 800, Chemistry Unit, latest revision

Operation of Rockwell Hardness Testers, Metallurgy Manual Metal 701, Chemistry Unit, latest revision

Operation of Microhardness Testers, Metallurgy Manual Metal 702, Chemistry Unit, latest revision

Operation of the Instron Model 3382 Universal Testing Machine, Metallurgy Manual Metal 703, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Balances, Instrument Operations Systems Support, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Micrometers and Calipers, Instrument Operations Systems Support, Chemistry Unit, latest revision

Performance Monitoring Protocol (QA-QC) for Scanning Electron Microscope (SEM)-Energy Dispersive X-ray Spectrometer (EDS), Instrument Operations Systems Support, Chemistry Unit, latest revision

Rev. #	Issue Date	History
6	12/21/2018	Reformatted and expanded section 5. Added section 8c. Augmented section 9 to include specific instrument procedures. Made minor editorial corrections throughout document. Added references to section 15 and corrected revision information.
7	01/29/2019	Added definitions of ‘fracture fit’, ‘inclusion’, ‘exclusion’, and ‘inconclusive’ conclusions to section 11. Updated titles of referenced documents.
8	02/18/2020	Revised section 7 and added Appendix A. Expanded section 8 to refer to techniques that use equipment already listed in section 5 and add minimization of destructive testing.

Approval Redacted - Signatures on File

Metallurgy Technical Leader: Date: 02/13/2020

Chemistry Unit Chief: Date: 02/13/2020

Appendix A: *Hypergeometric Table*

The hypergeometric table listed below shows the minimum number of samples that need to be analyzed (and yield consistent results) to obtain a 95% confidence level that at least 90% of the population contains a given substance.

Total Number of Units	Number of Units to be Sampled
1-10	All (no inferences)
11-13	10
14	11
15-16	12
17	13
18	14
19-24	15
25-26	16
27	17
28-35	18
36-37	19
38-46	20
47-48	21
49-58	22
59-77	23
78-88	24
89-118	25
119-178	26
179-298	27
299-1600	28
more than 1600	29